# Micro-Pak Enhanced Packaging Sheets: Enforceable Analytical Method

**DATA REQUIREMENTS:**

OPPTS 830.1800

**AUTHOR:**

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**STUDY COMPLETION DATE:**

~~11/29/12~~ 2/1/13 - KSB

**PERFORMING LABORATORY:**

Chemir Analytical Services

**LABORATORY TEST IDENTIFICATION:**

Sodium Chlorite by XRF

**DATA SUBMITTER:**

Micro-Pak Ltd.

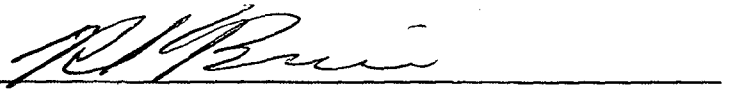
## STATEMENT OF DATA CONFIDENTIALITY CLAIMS

No claim of confidentiality, on any basis whatsoever, is made for any information contained in this document. I acknowledge that information not designated as within the scope of FIFRA Section 10(d)(1)(A), (B), or (C) and which pertains to a registered pesticide is not entitled to confidential treatment and may be released to the public, subject to the provisions regarding disclosure to multinational entities under FIFRA 10(g).

Company: **Micro-Pak Ltd.**

Company Agent: Robert S. Brennis
Brennis Consulting Services LLC

Title: Study Submitter and Agent for
Micro-Pak Ltd.

Signature: 

Date: 2/4/2013

## GOOD LABORATORY PRACTICE COMPLIANCE STATEMENT

This report only concerns the development of an enforceable analytical method for this product. There are no Good Laboratory Practice Standards (GLPS) specifically identified for the development of this data. Therefore, the Good Laboratory Practice Standards (GLPS), as specified in 40 CFR Part 160, are not applicable.

Author: _____ Date: 2-1-13

Name of Signer: John P. Simpson

Name of Company: Chemir Analytical Services

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Agent for Micro-Pak Ltd.

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Date: 2/1/13

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An analytical method to verify the certified limits for the total amount of Sodium Chlorite in the Enhanced Packaging Sheets is presented below. This method uses X-Ray Fluorescence (XRF) to quantify the amount of chlorine present in Micro-Pak Enhanced Packaging Sheets. The sodium chlorite concentration is then calculated from this determined chlorine value.

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## Quality Control Procedure

### Method Introduction

X-ray Fluorescence (XRF) is a non-destructive technique that can identify and quantify the elemental constituents of a sample using the secondary fluorescence signal produced by irradiation with high energy x-rays. This analysis utilized a wavelength dispersive spectrometer (WDXRF) that is capable of detecting elements from atomic number (Z) 4 (beryllium) through atomic number 92 (uranium) at concentrations from the low parts per million (ppm) range up to 100% by weight.

### List of Chemical Reagents

No chemicals were used during this analysis.

### List of Materials

A scalpel

### Instruments

The following comprises the instruments used during this procedure and all instrumental settings:

A Rigaku Primus II WDXRF with the following settings as summarized in table one.

Table 1: WDXRF Instrument parameters

| Parameter | Setting |
|---|---|
| X-Ray Source | Rhodium x-ray tube |
| X-Ray tube Power | 3.6 kW |
| Element Specific Tube Setting | 30kV, 120 mA |
| Scan Range | 90-96° 2θ |
| Crystal | Ge |
| Atmosphere | Vacuum |
| Analysis Area | 20 mm diameter |

## Standard Preparation and Calibration

The fundamental parameters approach was used for quantitation during the analysis. As a result, no standard is needed. The fundamental parameters approach uses x-ray physics coupled with established sensitivity factors for pure elements. The sensitivity factors for the spectrometer are established using pure element standards by the instrument manufacturer.

## Sample Preparation

Sections of the Micro-Pak Enhanced Packaging Sheets are cut and folded over multiple times to create a film stack 12 sheets thick. The stacking is necessary in order to present a sample of "infinite" thickness for the bulk measurement of chlorine in the polymer. A thickness of ~80µm is required.

The measurement area of the method was approximately 30mm by 30mm and was carried out in a vacuum. The sample was prepared three times with each preparation analyzed in triplicate.

Data was collected in both scanned and fixed angle modes. In normal scanning mode analysis, the detector is rotated through the full range of dispersion to collect the diffracted x-ray signals coming from the sample and spectra are generated. In a fixed angle analysis the detector dwells at a specific angle for an extended period while collecting a signal only from the peak maximum and from the adjacent background. Fixed angle analysis significantly improves sensitivities. Since a fixed angle analysis collects intensities from only two points (peak maximum and baseline) no spectra are created. The sample was prepared three times with each preparation analyzed in triplicate. From the determined chloride concentration, the amount of sodium chlorite present in the same was calculated using the assumption that all chloride present was in the form of sodium chlorite (see calculation below).

## Calculation of Weight Percent Active

From the determined chloride concentration, the amount of sodium chlorite present in the same was calculated using the assumption that all chloride present was in the form of sodium chlorite (see calculation below).

$$Chlorite\ Conc.\ in\ Sample\ (mass\%) = Chlorine\ Conc.\ (\%) \times \frac{Sodium\ Chlorite\ mass\ (90.442\ g/mol)}{Chlorine\ mass\ (34.45\ g/mol)}$$

## Method Performance

The reported values of the analysis of one sample are present in Table 2. The relative standard deviation over all measurements was below 5 % with an average sodium chlorite concentration of 0.143% by mass.

Table 2: Sodium Chlorite concentration

| *Preparation.* | *Run* | *Sodium Chlorite Conc. (%)* | *Average* | *%RSD* |
|---|---|---|---|---|
| 1 | *1* | 0.138 | | |
| 1 | *2* | 0.138 | | |
| 1 | *3* | 0.138 | 0.138 | 0.00 |
| 2 | *4* | 0.140 | | |
| 2 | *5* | 0.143 | | |
| 2 | *6* | 0.140 | 0.141 | 1.04 |
| 3 | *7* | 0.158 | | |
| 3 | *8* | 0.148 | | |
| 3 | *9* | 0.145 | 0.151 | 4.48 |
| | ***Average*** | 0.143 | | |
| | ***%RSD*** | 4.67 | | |